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National Food Safety Standard-Erythrosine Aluminum Lake

Report Categories:

FAIRS Subject Report

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Report Highlights:
On May 5, 2010, China notified the WTO of National Food Safety Standard: Food Additives – Erythrosine Aluminum Lake as SPS/N/CHN/277. This measure applies to the production, circulation, supervision and management of the food additive Erythrosine Aluminum Lake. It specifies the scope, requirements and testing methods. The date for submission of final comments to China is May 20, 2010. The proposed date of entry is May 30, 2010. Contact information on where to send comments is inside the report. This report is an INFORMAL translation of this document.

Executive Summary:
On May 5, 2010, China notified the WTO of National Food Safety Standard: Food Additives – Erythrosine Aluminum Lake as SPS/N/CHN/277. This measure applies to the production, circulation, supervision and management of the food additive Erythrosine Aluminum Lake. It specifies the scope, requirements and testing methods. The date for submission of final comments to China is May 20, 2010. The proposed date of entry is May 30, 2010.

Comments can be sent to the China WTO SPS Enquiry Point at: SPS@aqsiq.gov.cn.

This report contains an UNOFFICIAL translation of National Standard on Determination of Erythrosine Aluminum Lake in Foods.

General Information:
BEGIN TRANSLATION

GB National Food Safety Standard

GB 17512.2-XXX

Food Additive - Erythrosine Aluminum Lake

National Food Safety Standard

(Draft for Comment)

Issued on xx-xx-xxxx

Implemented on xx-xx-xxxx

Issued by the Ministry of Health

of the People's Republic of China

Foreword

This Standard is modified in relation to "Food Red No. 3" in Japan's Specifications and Standards for Food Additives (Edition 8).

Main technical differences between this Standard and "Food Red No. 3" in Japan's Specifications and Standards for Food (Edition 8) are as follows:

-- Test method for subsidiary colors is spectrophotometric method after thin-layer chromatography and elution treatment, and the requirement is not more than 1.5% in this Standard; while no exact requirement is stipulated and spotting method is taken as test method in Japan's standard;

-- Arsenic requirement is not more than 3.0 (mg/kg) and test method is atomic absorption method in this Standard; while arsenic requirement is not more than 0.0004 % (based on As2O3) and limit colorimetric method is test method in Japan's standard;

-- Requirement for heavy metals (based on Pb) is modified into requirement for lead content and atomic absorption method is taken as test method in this Standard; while requirement is still for heavy metals (based on Pb) and test method is limit colorimetric method in Japan's standard.

This Standard supersedes GB 17512.2-1998 Food Additive - Erythrosine Aluminum Lake.

Compared with GB 17512.2-1998, this Standard has main changes as follow:

-- modifying identification method;

-- adding permissible difference for parallel determinations by spectrophotometric colorimetric method;

-- cancelling requirements for chloride (based on NaCl) and sulfate (based on Na2SO4);

-- modifying chemical half-limit method for arsenic test into atomic absorption method;

-- modifying requirement for heavy metals (based on Pb) into control requirement of lead and test method into atomic absorption method;

-- adding control requirement and test method for zinc;

-- modifying barium determination method into limit colorimetric method for barium sulfate precipitate.

Annex A of this Standard is normative.

This Standard supersedes the following previous edition:

-- GB 17512.2-1998

National Food Safety Standards

Food Additive – Erythrosine Aluminum Lake

1 Scope

This Standard is applicable to quality control of food additive erythrosine and of erythrosine aluminum lake products produced by aluminum hydroxide action.

2 Normative references

Documents referenced in this Standard are indispensable for the application of this Standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

3 Molecular formula and relative molecular mass

Molecular formula: C20H6I4Na2O5 •H2O

Relative molecular mass: 897.87 (based on 2007 International Relative Atomic Mass)

4 Technical requirements

Technical requirements of erythrosine aluminum lake shall be in accordance with Table 1.

Table 1 Technical requirements of erythrosine aluminum lake

Items	Requirement	Test method

Appearance	Red powder	Visual inspection under natural light
Erythrosine (based on sodium salt), *w*/%	≥10.0	A.3 in Annex A
Loss on drying, w/%	≤30.0	A.4 in Annex A
Insoluble matters in hydrochloric acid and ammonia, *w*/%	≤0.50	A.5 in Annex A
Subsidiary colors, *w* /%	≤1.5	A.6 in Annex A
Sodium iodide, *w* /%	≤0.2	A.7 in Annex A
Arsenic, mg/kg	≤3.0	A.8 in Annex A
Lead, mg/kg	≤10.0	A.9 in Annex A
Zinc, mg/kg	≤50.0	A.10 in Annex A
Barium (based on Ba), *w* /%	≤0.05	A.11 in Annex A

Annex A

(Normative)

Test Method

A.1 General requirements

Reagents and water used in this Standard, unless otherwise stated, are analytically pure reagents and grade III water specified in GB/T 6682-2008. Standard solution, impurity standard solution, preparations and products used in the tests, unless otherwise stated, shall be prepared and calibrated according to requirements of GB/T 601, GB/T 602 and GB/T 603. Test results shall be judged in accordance with 4.3.3 Round-off comparison method in GB/T 8170-2008.

A.2 Identification

A.2.1 Reagents and solutions

a) Sulfuric acid;

b) Hydrochloric acid solution: 1 + 3;

c) Sodium hydroxide solution: 90 g/L;

d) Ammonium acetate solution: 1.5 g/L;

e) Activated carbon.

A.2.2 Apparatus

a) Spectrophotometer;

b) Cuvette: 10 mm.

A.2.3 Identification method

Weigh about 0.1 g of the sample, add 5 mL of sulfuric acid, and shake frequently in water bath. Heat for about 5 min, the solution shall develop orange red. After cooling, measure 2 - 3 drops of supernatant, and add 5 mL of water, the solution shall develop red.

Weigh about 0.1 g of the sample, add 5 mL of sodium hydroxide solution, heat in water bath for dissolving, and add ammonium acetate solution to make 100 mL, perform centrifugal separation when the resulting solution is turbid, then measure 1 mL - 10 mL of the solution, add ammonium acetate solution to make 100 mL, and make determined absorbance within 0.2 - 0.7. The maximum absorption wavelength of the solution is 526 nm ± 2 nm.

Weigh about 0.1 g of the sample; add 10 mL of hydrochloric acid solution, heat in water bath to make most of the sample dissolve. Add 0.5 g of activated carbon, thoroughly shake up and filter. Measure colorless filtrate and add sodium hydroxide solution for neutralization, and the resulting solution shall produce aluminum salt reaction.

A.3 Determination of erythrosine aluminum lake

A.3.1 Method summary

Dissolve treated sample and erythrosine with known content in ammonium acetate solution respectively, determine respective absorbance values at maximum absorbance wavelength, and then calculate contents.

A.3.2 Reagents and solutions

a) Ammonia solution: 1 + 3;

b) Ammonium acetate solution: 1.5 g/L;

c) Erythrosine standard substance: ≥ 85.0% (mass fraction, determine according to A.3.1 in this Standard).

A.3.3 Apparatus and instruments

a) Spectrophotometer;

b) Cuvette: 10 mm.

A.3.4 Preparation of erythrosine standard solution

Weigh about 0.25 g of erythrosine standard substance (accurate to 0.0001 g), dissolve in a proper amount of ammonium acetate solution, transfer to a 1000 mL volumetric flask, add ammonium acetate solution to dilute to volume, and shake up. Pipette 10 mL of the resulting solution to a 500 mL volumetric flask, add ammonium acetate solution to dilute to volume, and shake up.

A.3.5. Preparation of erythrosine aluminum lake sample solution

Weigh about 0.5 g of the sample (accurate to 0.0001 g), transfer to a 250 mL volumetric flask, add 150 mL of ammonia solution, constantly stir for dissolving, transfer to a 500 mL volumetric flask, dilute to volume with water, and shake up. Pipette 10 mL of the resulting solution to a 500 mL volumetric flask, add ammonium acetate solution to dilute to volume, and shake up.

A.3.6. Determination procedures

Place erythrosine standard solution and erythrosine aluminum lake sample solution in 10 mm cuvettes respectively, determine respective absorbance values at maximum absorption wavelength by spectrophotometer, and take ammonium acetate solution as reference solution.

A.3.7 Result calculation

Erythrosine aluminum lake is calculated according to formula (A.1) based on mass fraction w1 and its value is expressed in %:

$$w_1 = \frac{A_1(m_0/1000\times 10/500)}{A_0(m_1/500\times 10/500)}\times w_0 \qquad (A.1)$$

where:

A1 --absorbance value of erythrosine aluminum lake sample solution;

m0--value of mass of erythrosine standard substance; expressed in g;

--content of erythrosine standard substance; expressed in % (mass fraction);

A0--absorbance value of erythrosine standard solution;

m1--value of mass of erythrosine aluminum lake sample; expressed in g.

Calculation result is rounded to 0.1.

A.3.8 Permissible difference

Absolute difference between two parallel determination results is not more than 1.0 % (mass fraction). Arithmetic mean is taken as determination result.

A.4 Determination of loss on drying

A.4.1 Determination procedures

Weigh about 2 g of the sample (accurate to 0.001 g), place in a weighing bottle made to constant weight, and bake the weighing bottle in 135 ℃ constant temperature oven to constant weight.

A.4.2 Result calculation

Loss on drying is calculated according to formula (A.2) based on mass fraction w2 and its value is expressed in %:

$$w_2 = \frac{m_2 - m_3}{m_2} \times 100 \quad (A.2)$$

where:

m2--value of mass of the sample before drying, expressed in g;

m3--value of mass of the sample after drying to constant weight, expressed in g.

Calculation result is rounded to 0.1.

A.4.3 Permissible difference

Absolute difference between two parallel determination results is not more than 0.2% (mass fraction). Arithmetic mean is taken as determination result.

A.5 Determination of insoluble matters in hydrochloric acid and ammonia

A.5.1 Reagents and solutions

a) Hydrochloric acid;

b) Hydrochloric acid solution: 3 + 7;

c) Ammonia solution: 4 + 96;

d) Silver nitrate solution: c(AgNO3) = 0.1 mol/L.

A.5.2 Apparatus

a) Sintered glass crucible: G4, aperture: 5 μm - 15 μm;

b) Constant temperature oven.

A.5.3 Determination procedures

Weigh about 2 g of the sample (accurate to 0.001 g), place in a 600 mL beaker, add 20 mL of water and 20 mL of hydrochloric acid, stir thoroughly, add 300 mL of hot water, stir fully, cover watch glass, heat the beaker in 70 ℃ - 80 ℃ water bath for 30 min, cool, filter by G4 sintered glass crucible baked at 135 ℃ to constant weight, wash insoluble matters in the beaker with about 30 mL of water to G4 sintered glass crucible till the solution is colorless, wash insoluble

matters with 100 mL of ammonia solution and 10 mL of hydrochloric acid solution in order, and wash with water till no white precipitate is detected in the solution by silver nitrate solution, and then bake in a 135 ℃ constant temperature oven to constant weight.

A.5.4 Result calculation

Insoluble matters in hydrochloric acid and ammonia are calculated according to formula (A.3) based on mass fraction w3 and their value is expressed in %:

$$w_3 = \frac{m_4}{m_5} \times 100 \quad \text{.....(A.3)}$$

where:

m4--value of mass of water insoluble matters after drying; expressed in g;

m5--value of sample mass; expressed in g.

Calculation result is rounded to 0.01.

A.5.5 Permissible difference

Absolute difference between two parallel determination results is not more than 0.05% (mass fraction). Arithmetic mean is taken as determination result.

A.6 Determination of subsidiary colors

A.6.1 Method summary

Separate and elute components by paper chromatography, and determine by spectrophotography.

A.6.2 Reagents and solutions

a) Absolute ethyl alcohol;

b) N-butyl alcohol;

c) Acetone solution: 1 + 1;

d) Ammonia solution: 4 + 96;

e) Sodium bicarbonate solution: 4 g/L.

A.6.3 Apparatus and instruments

a) Spectrophotometer;

b) Chromatography filter paper: No. 1 medium speed, 150 mm × 250 mm;

c) Chromatography tank: φ240 mm × 300 mm;

d) Micro sample injector: 100 μL;

e) Nessler tube: 50 mL, having ground glass stopper;

f) Sintered glass funnel: G3; aperture: 15 μm - 40 μm;

g) 50 mm cuvette;

h) 10 mm cuvette.

A.6.4 Determination procedures

A.6.4.1 Conditions for paper chromatography

a) Developing solvent: n-butyl alcohol + absolute ethyl alcohol + ammonia solution = 6 + 2 + 3;

b) Temperature: 20 ℃ - 25 ℃.

A.6.4.2 Preparation of sample solution

Weigh about 2 g of sample (accurate to 0.001 g), place in a beaker, add a proper amount of water and 50 mL of sodium hydroxide solution, heat for dissolving, transfer to a 100 mL volumetric flask, dilute to volume and shake up for use. Concentration of the sample solution is 2 %.

A.6.4.3 Preparation of sample eluate

Pipette 100 μL of sample solution by micro sample injector, evenly inject on a baseline 25 mm away from bottom edge of filter paper to form a straight line with width not more than 5 mm and the length of 130 mm on the filter paper, blow dry with an air blower. Develop the filter paper in the chromatography tank with pre-prepared developing solvent. Immerse the bottom edge of filter paper 10 mm below the developing solvent front line of the developing solvent rises to 150 mm or subsidiary colors are separated to satisfaction, take out chromatography filter paper, and blow dry with cold air.

Develop blank filter paper under the same condition. The blank filter paper shall be cut from adjacent part of the same filter paper as the filter paper used in previous developing procedures.

Schematic diagram of paper chromatography of subsidiary colors is shown in Fig. A.1.

Cut filter paper of various developed subsidiary colors and parts of filter paper corresponding to subsidiary colors on the blank filter paper at the same size, cut into 5 mm × 15 mm strips, place in 50 mL Nessler tubes respectively, accurately add 5 mL of acetone solution and shake for 3 min - 5 min, accurately add 20 mL of sodium hydrocarbonate solution again and shake thoroughly, and filter the resulting solution by G3 sintered glass funnel respectively, the resulting filtrate must be clear and free of suspension. Obtain eluates of subsidiary colors and blank solution respectively. Use 50 mm cuvette to measure absorbance of eluates of subsidiary colors by the spectrophotometer at the maximum absorption wavelength of subsidiary colors.

Use mixture of 5 mL of acetone solution and 20 mL of sodium hydrocarbonate solution as reference solution when measuring absorbance by the spectrophotometer.

A.6.4.4 Preparation of standard solution

Pipette 6 mL of 2 % sample solution to a 100 mL volumetric flask, dilute to volume, and shake up. The resulting solution is standard solution.

A.6.4.5 Preparation of standard eluate

Pipette 100 μL of standard solution by the micro sample injector, evenly inject on a baseline 25 mm away from the bottom edge of filter paper, and blow dry with an air blower. Put filter paper in a chromatography tank with pre-prepared developing solvent for developing, take out and cool the filter paper with cold air after front line of developing solvent rises to 40 mm, cut all parts with developed colors and extract by the method in A.6.4.3 of this Standard to obtain standard eluate. Use 10 mm cuvette to measure absorbance value at the maximum absorption wavelength.

Meanwhile, develop blank filter paper under the same conditions and measure absorbance value of eluate according to the same procedures.

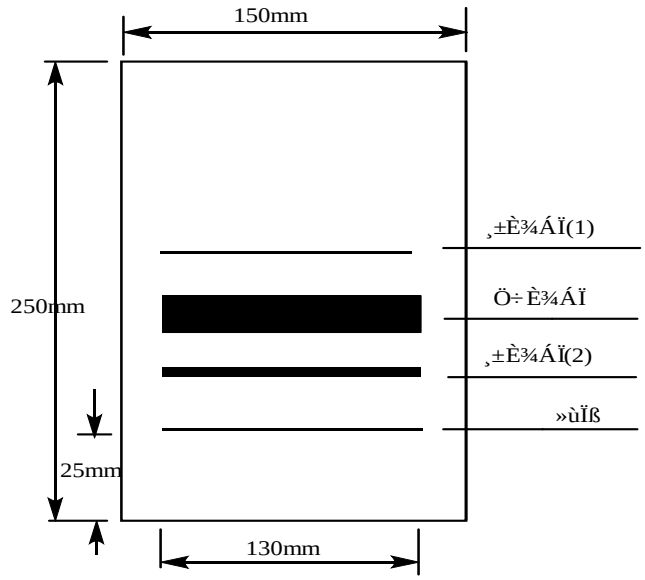

Fig. A.1 Schematic diagram of paper chromatography of subsidiary colors

A.6.4.6 Result calculation

Subsidiary colors are calculated according to formula (A.4) based on mass fraction w4 and their value is expressed in %:

$$w_4 = \frac{[(A_1 - b_1) + \cdots\cdots + (A_n - b_n)]5}{(A_s - b_s)(100/6)} \times S \qquad (A4)$$

where:

A1…, An--absorbance values of eluates of subsidiary colors measured at 50 mm beam path distance;

b1…, bn--absorbance values of control blank eluates of subsidiary colors measured at 50 mm beam path distance;

As--absorbance value of standard eluate measured at 10 mm beam path distance;

bs--absorbance value of standard control blank eluate measured at 10 mm beam path distance;

5--ratio of being converted into 10 mm beam path distance;

100/6--ratio of standard eluate converted into 2 % sample solution;

S--sample content; expressed in % (mass fraction).

Calculation result is rounded to 0.1.

A.6.4.7 Permissible difference

Absolute difference between two parallel determination results is not more than 0.2 % (mass fraction). Arithmetic mean is taken as determination result.

A.7 Determination of sodium iodide

A.7.1 Method summary

Titrate content of sodium iodide in sample solution with silver nitrate standard titration solution by potentiometric titration.

A.7.2 Reagent

Silver nitrate standard titration solution: c(AgNO3) = 0.001 mol/L.

A.7.3 Apparatus and instruments

a) Digital millivoltmeter;

b) Iodine ion selective electrode;

c) Reference electrode;

d) Electromagnetic stirrer.

A.7.4 Preparation of sample solution

Weigh about 2.0 g of sample (accurate to 0.0001 g), place in a beaker, add accurately measured 100 mL of water, and stir with electromagnetic stirrer for dissolving, put dry filter paper in funnel funnel of sintered glass crucible for filtering, and use the resulting solution as sample solution.

A.7.5 Determination procedures

Insert iodine ion selective electrode and reference electrode into test solution, adjust readings of millivoltmeter, and titrate with silver nitrate standard titration solution while stirring thoroughly. Titrate 0.5 mL at beginning of titration, and increase titer gradually, observe electric potential change after each titration and record reading. Lower titer to 0.1 mL when closing to end point, draw a curve with electric potential millivoltmeter readings and titer volume of relevant silver nitrate standard titration solution, consider the maximum jump point of the curve as titration end point and obtain volume of silver nitrate standard titration solution.

A.7.6 Result calculation

Sodium iodide is calculated according to formula (A.5) based on mass fraction w5 and its value is expressed in %:

$$w_5 = \frac{c\ (V/1000\ )M}{m_6} \times 100 \qquad (A.5)$$

where:

c--accurate value of concentration of silver nitrate standard titration solution; expressed in mol/L;

V--accurate value of volume of silver nitrate standard titration solution consumed for titrating sample; expressed in mL;

M--value of molar mass of sodium iodide; expressed in g/mol [M(NaI) =149.89];

m6--value of sample mass; expressed in g.

Calculation result is rounded to 0.1.

A.8 Determination of arsenic

A.8.1 Method summary

Digest erythrosine aluminum lake by wet method, prepare into sample solution, and determine arsenic content by atomic absorption spectrometry.

A.8.2 Reagents and solutions

a) Nitric acid;

b) Sulfuric acid solution: 1 + 1;

c) Nitric acid-perchloric acid mixed solution: 3 + 1;

d) Arsenic (As) standard solution: after preparation and calibration in accordance with GB/T 602, dilute and prepare into three standard solutions with relevant arsenic concentrations

according to requirements of used apparatus;

e) Sodium hydroxide solution: 1 g/L;

f) Sodium borohydride solution: 8 g/L(solvent is 1 g/L sodium hydroxide solution);

g) Hydrochloric acid solution:1 + 10;

h) Potassium iodide solution: 200 g/L.

A.8.3 Apparatus

Atomic absorption spectrometer

Reference conditions of apparatus: analysis line wavelength of arsenic hollow cathode lamp: 193.7 nm; slit: 0.5 nm - 1.0 nm; lamp current: 6 mA-10 mA;

Flow rate of carrier gas: 250 mL/min, argon gas ;

Temperature of atomizer: 900 ℃.

A.8.4 Determination procedures

A.8.4.1 Sample digestion

Weigh about 1.0 g of sample (accurate to 0.001 g), place in a 250 mL conical or round bottomed flask, add 10 mL - 15 mL of nitric acid and 2 mL of sulfuric acid, shake up, and heat with low fire to remove nitrogen dioxide gas, stop heating when solution develops brown, cool naturally, add 5 mL of nitric acid-perchloric acid mixed solution, heat with strong fire till the solution is colorless and transparent or yellowish. In case of opaque solution, cool naturally, add 5 mL of nitric acid-perchloric acid mixed solution again, keep heating till solution is colorless and transparent or yellowish and produces white smoke (avoid carbonization due to burning out), stop heating, cool naturally, add 5 mL of water, heat to boil to remove residual acid-perchloric acid (add water to boil again when necessary). Keep heating to produce white smoke and keep 10 min, cool naturally, transfer to a 100 mL volumetric flask (filter in case of turbidity, precipitate and mechanical impurities in solution), and dilute to volume with hydrochloric acid solution.

Meanwhile, prepare blank solution by the same method.

A.8.4.2 Determination

Measure 25 mL of digested sample solution to a 50 mL volumetric flask, add 5 mL of potassium iodide solution, dilute to volume with hydrochloric acid solution, shake up, and stand for 15 min.

Meanwhile, prepare blank test solution with blank solution by the same method.

Turn on the apparatus, after the apparatus and the arsenic hallow cathode lamp are fully preheated and baseline is stable, use sodium borohydride solution as hydride reducing agent and inject standard blank solution, standard solution, sample blank test solution and sample solution in order according to computer instruction. After test, computer can automatically generate working curve and arsenic concentration of sample solution after deducting sample blank solution, automatically calculate arsenic content after inputting sample information (name, weight, dilution volume, etc.).

A.8.4.3 Permissible difference

Absolute difference between two parallel determination results is not more than 0.1 (mg/kg). Arithmetic mean is taken as determination result.

A.9 Determination of lead

A.9.1 Method summary

Digest erythrosine aluminum lake by wet method, prepare into sample solution, and determine

lead content by atomic absorption spectrometry.

A.9.2 Reagents and solutions

a) Lead (Pb) standard solution: after preparation and calibration in accordance with GB/T 602, dilute and prepare into three standard solutions with relevant lead concentrations according to requirements of used apparatus;

b) Sodium hydroxide solution: 1 g/L;

c) Sodium borohydride solution: 8 g/L (solvent is 1 g/L sodium hydroxide solution);

d) Hydrochloric acid solution: 1 + 10.

A.9.3 Apparatus

Atomic absorption spectrometer

Reference conditions of apparatus: Method 3 - Flame atomic absorption spectrometry in GB 5009.12.

A.9.4 Determination procedures

The sample solution and blank solution in A.8.4.1 of this Standard can be directly used.

Operate according to Method 3 - Flame atomic absorption spectrometry in GB 5009.12.

A.9.5 Permissible difference

Absolute difference between two parallel determination results is not more than 1.0 (mg/kg). Arithmetic mean is taken as determination result.

A.10 Determination of zinc

A.10.1 Method summary

Digest erythrosine aluminum lake by wet method, prepare into sample solution and determine zinc content by atomic absorption spectrometry.

A.10.2 Reagents and solutions

a) Zinc (Zn) standard solution: after preparation and calibration in accordance with GB/T 602, dilute and prepare into three standard solutions with relevant zinc concentrations according to requirements of used apparatus;

b) Sodium hydroxide solution: 1 g/L;

c) Sodium borohydride solution: 8 g/L(solvent is 1 g/L sodium hydroxide solution);

d) Hydrochloric acid solution: 1 + 10.

A.10.3 Apparatus

Atomic absorption spectrometer

Reference conditions of apparatus: Method 1 - Atomic absorption spectrometry in GB 5009.14.

A.10.4 Determination procedures

The sample solution and blank solution in A.8.4.1 of this Standard can be directly used.

Determine according to Method 1 - Atomic absorption spectrometry in GB 5009.14.

A.10.5 Permissible difference

Absolute difference between two parallel determination results is not more than 5.0 (mg/kg). Arithmetic mean is taken as determination result.

A.11 Determination of barium (based on Ba)

A.11.1 Method summary

Digest erythrosine aluminum lake by dry method, prepare into sample solution, and perform turbidity limit test on barium sulphate after comparison with barium standard solution.

A.11.2 Reagents

a) Sulfuric acid;

b) Anhydrous sodium carbonate;

c) Hydrochloric acid solution: 1 + 3;

d) Sulfuric acid solution: 1 + 19;

e) Barium standard solution: dissolve 177.9 mg of barium chloride ($BaCl_2 \bullet 2H_2O$) in water and dilute to 1000 mL. Each mL of the solution contains 0.1 g of barium (0.1 mg/mL).

A.11.3 Preparation of sample solution

Weigh about 1 g of the sample (accurate to 0.001 g), place in a platinum crucible or ceramic crucible, add a little sulfuric acid to wet the sample, and slowly heat to almost fully carbonize it at low temperature, cool naturally, add 1 mL of sulfuric acid, and slowly heat until almost no sulfuric acid vapor produces. Put the crucibles in a high temperature furnace and ignite at 800 ℃ for 3 h, cool down, add 5 g of anhydrous sodium carbonate and thoroughly mix, cover the crucibles and put in the high temperature furnace, ignite at 860 ℃ for 15 min, cool down, add 20 mL of water, and heat in water bath to dissolving melt. Cool down, filter, and wash residue on filter paper with water until the cleaning solution does not produce sulfate action. Transfer the residue on filter paper and filter paper together to a beaker, add 30 mL of hydrochloric acid solution, shake up and boil. Cool down, filter, and wash residue on filter paper with 10 mL of water. Mix cleaning solution and filtrate, and vaporize to dry in water bath. Add 5 mL of water to make residue dissolve, filter if necessary, add 0.25 mL of hydrochloric acid solution, mix thoroughly, and add water to make 25 mL. The resulting solution is used as sample solution.

A.11.4 Preparation of standard turbidimetric solution

Measure 5 mL of barium standard solution, add 0.25 mL of hydrochloric acid solution, add water to 25 mL, and use the resulting solution as standard turbidimetric solution.

A.11.5 Determination procedures

Add 1 mL of sulfuric acid solution to sample solution and standard turbidimetric solution respectively and stand for 10 min. It is acceptable if turbidity of the sample solution is not more than that of standard turbidimetric solution.

END TRANSLATION